



IN THE UNITED STATES OF PATENT AND TRADEMARK OFFICE

In re the application

Sakashita et. al.

Group Art Unit 1713

Serial Number: 10/730,887

Examiner: Dr. Kelechi C. Egwim

Filed: December 10, 2003

For: PROCESSING AID FOR VINYL CHLORIDE RESIN AND VINYL CHLORIDE

RESIN COMPOSITION

DECLARATION UNDER 37 CFR 1.132

Commissioner for Patents

Washington, D. C. 20231

Sirs:

Mitsutaka Sato residing at 10-35-604, Yamashitacho, Akashi-shi, Hyogo, Japan duly deposes and says:

1. That he graduated from Department of Applied Chemistry, Faculty of Engineering, Muroran Institute of Technology, Hokkaido, Japan in the year 1997, he received the degree of Mater of Applied Chemistry from Muroran Institute of Technology graduate school, Hokkaido, Japan in the year 1999;
2. That since 1999, he has been employed in KANEKA CORPORATION;
3. That from 1999 he has been engaged in research and development of processing aid for polyvinyl chloride resins;
4. That he has read and is familiar with the instant application for United States Letters Patent and Office Action thereto mailed April 17, 2007; and
5. That he has made experiments in order to prove that a processing aid for a vinyl chloride resin of the present application is more excellent than those disclosed in the cited references particularly because of the

criticality of the specific viscosity at the first step and the second step.

6. Experiments were carried out by the following procedure.

**Experiment**

<Sample 1>

0.7 Part by weight of sodium dioctyl sulfosuccinate previously dissolved in water was charged in an 8 liter reactor equipped with a stirrer, and then water was added thereto in such an amount that the total water content including water contained in sub-materials to be added later on was 200 parts by weight. Oxygen in the space of the reactor and in water was removed by introducing nitrogen gas in the liquid part of the reactor, and then the content was heated up to 70°C with stirring.

Then, a monomer mixture (A) containing 60 parts by weight of methyl methacrylate (MMA) and 20 parts by weight of butyl acrylate (BA) was added one and the same, and then 0.04 part by weight of potassium persulfate was further added, followed by continuing stirring for 1 hour to complete substantially the polymerization.

Thereafter, a monomer mixture (B) containing 6 parts by weight of MMA and 14 parts by weight of BA was dropped by about 30 parts by weight per 1 hour. After dropping, the content was kept at 70°C for 90 minutes, followed by cooling, whereby a latex was obtained.

A polymerization conversion rate of the resulting latex was 99.4 %. The obtained latex was solidified by salting-out. After treatment by increasing a temperature up to 90°C, the resultant was filtered by a centrifugal dehydrator. A dehydrated cake of the obtained two-step polymer mixture washed with water of about the same weight as the polymer mixture and dried at 50°C for 15 hours by a parallel flow drier to give the sample (1) as white powder.

(Specific viscosity)

0.1 Gram of the sample (1) was dissolved in 100 ml of chloroform, and kept at a constant temperature in a water bath of 30°C, and then a specific viscosity in chloroform was measured by Ubbelohde's viscometer.

(Foamability)

To 100 parts by weight of polyvinyl chloride (an average polymerization degree of 680) were incorporated 2.0 parts by weight of isooctyl mercaptoacetate, 2.0 parts by weight of titanium oxide, 6.0 parts by weight of calcium carbonate, 0.6 part by weight of calcium stearate, 0.1 part by weight of hydroxy stearic acid (Cognis, LOXIOL G-21), 0.9 part by weight of a fatty acid ester (Cognis, LOXIOL G-60) and 0.6 part by weight of polyethylene oxide wax (Allied-Signal, A-C629A) and the resultant was kneaded by a Henschel mixer. The resultant was heated up to an inner temperature of 110°C, and cooled to give a powdery compound. To the compound was further incorporated 4 parts by weight of sodium bicarbonate to give a powdery compound. The resultant was molded at C1/C2/C3/D=170°C /175°C /180°C /185°C under 30 rpm by a single axis extruder (TOYO SEIKI Co., Ltd., 5 mm round die) A specific gravity of the resulting a round stick shape molded article was evaluated. As the specific gravity of the molded article became lower, a foaming rate became high, namely foamability became good. Formulations and the results were shown in Table 2 and Figure 2.

**<Sample (2)>**

In order to obtain samples having different specific viscosity, addition of the monomer mixture (B) was conducted one and the same in place of stepwise addition to give sample (2). The obtained result was shown in Table 2 and Figure 2.

**<Sample (3)>**

0.1 Part by weight of sodium dioctyl sulfosuccinate previously dissolved in water was charged in an 8 liter reactor equipped with a stirrer, and then water was added thereto in such an amount that the total water content including water contained in sub-materials to be added later on was 200 parts by weight. Oxygen in the space of the reactor and in water was removed by introducing nitrogen gas in the liquid part of the reactor, and then the content was heated up to 70°C with stirring.

Then, 0.01 part by weight of potassium persulfate was added to the reactor, a monomer mixture (A) containing 60 parts by weight of methyl methacrylate (MMA) and 20 parts by weight of butyl acrylate (BA) was dropped by about 30 parts by weight per one hour, and further each 0.3 part by weight of sodium dioctyl sulfosuccinate was added at the time of 1 hour after and at the time of 2 hour after. After dropping of the monomer mixture (A), stirring was continued for 1 hour and a

half to complete substantially the polymerization.

Thereafter, a monomer mixture (B) containing 6 parts by weight of MMA and 14 parts by weight of BA was added one and the same, and the content was kept at 70°C for 90 minutes, followed by cooling, whereby a latex was obtained similarly to the sample (1). The result was shown in Table 1 and Figure 1.

<Sample (4)>

0.7 Part by weight of sodium dioctyl sulfosuccinate previously dissolved in water was charged in an 8 liter reactor equipped with a stirrer, and then water was added thereto in such an amount that the total water content including water contained in sub-materials to be added later on was 200 parts by weight. Oxygen in the space of the reactor and in water was removed by introducing nitrogen gas in the liquid part of the reactor, and then the content was heated up to 70°C with stirring.

Then, a monomer mixture (A) containing 60 parts by weight of methyl methacrylate (MMA) and 20 parts by weight of butyl acrylate (BA) was added one and the same, and then 0.04 part by weight of potassium persulfate was further added, followed by continuing stirring for 1 hour to complete substantially the polymerization.

Thereafter, a mixed solution of 0.01 part by weight of ethylenediamine sodium tetraacetate and 0.005 part by weight of ferrous sulfate monohydrate in 5 parts by weight of distilled water and a mixed solution of 0.3 part by weight of formaldehyde sodium sulfoxylate and 0.3 part by weight of dioctyl sodium sulfosuccinate in 10 parts by weight of distilled water were charged, and to the resultant dropped a monomer mixture (B) containing 6 parts by weight of MMA, 14 parts by weight of BA and 0.3 part by weight of t-butyl hydroperoxide. After dropping, the content was kept at 70°C for 90 minutes, followed by cooling, whereby a latex was obtained. Then the sample (4) was obtained after a similar manner to the sample 1. The obtained result was shown in Table 2 and Figure 2.

<Samples (5) to (8)>

In order to obtain samples having different specific viscosity, the samples (5) to (8) were obtained by using 0.003 to 0.06 part by weight of sodium persulfate and 0.01 to 0.1 part by weight of t-butyl hydroperoxide in place of those in the sample (4). The obtained result was shown in Table 1, Table 2, Figure 1 and Figure 2.

In the above Tables and Figures;

Table 1 and Figure 2 show foamabilities of the samples wherein the second step  $\eta$  sp was 0.6 or more and the first step  $\eta$  sp was varied from 0.54 to 0.86.

Table 2 and Figure 2 show foamabilities of the samples wherein the second step  $\eta$  sp was 0.7 or more and the first step  $\eta$  sp was varied from 0.31 to 0.81.

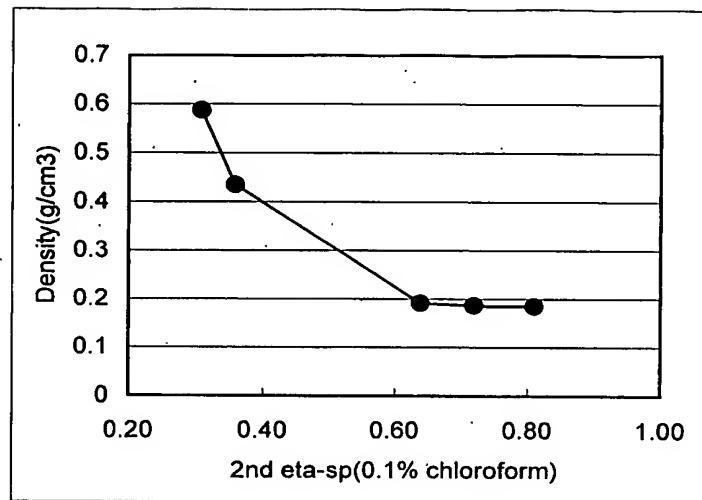
Table 1

Sample No.	(3)	(7)	(6)	(8)
pottassium persulfate (part)	0.01	0.05	0.04	0.01 5
t-butyl hydroperoxide (partA)	-	0.01	0.01	0.01
$\eta$ sp at the first step polymerization	0.54	0.62	0.72	0.86
$\eta$ sp of the second step polymerized mixture	0.61	0.60	0.64	0.63
foamabilityg/cm <sup>3</sup> )	0.50	0.36	0.19	0.18
latex particle size (Å)	1700	1500	1500	1600

Table 2

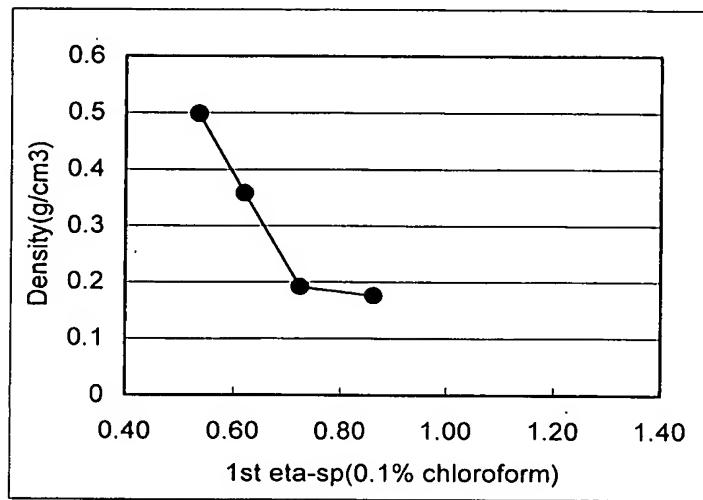
Sample No.	(4)	(5)	(6)	(1)	(2)
pottassium persulfate (part)	0.04	0.04	0.04	0.04	0.04
t-butyl hydroperoxide (partA)	0.3	0.1	0.01	-	-
$\eta$ sp at the first step polymerization	0.71	0.72	0.72	0.71	0.72
$\eta$ sp of the second step polymerized mixture	0.31	0.36	0.64	0.72	0.81
foamabilityg/cm <sup>3</sup> )	0.59	0.43	0.19	0.19	0.18
latex particle size (Å)	1500	1500	1500	1500	1500

Figure 1



relationship between  $\eta$  sp and density of the first step polymer

Figure 2



relationship between  $\eta$  sp and density of the second step polymer mixture

The undersigned declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed this 29 day of June, 2007

Mitsutaka Sato

by Mitsutaka Sato

We, the undersigned witnesses, hereby acknowledge that Mitsutaka Sato is personally known to us and did execute the foregoing Declaration in our presence on:

Date: June 29, 2007 Witness Hideyuki Sakurai

Date: June 29, 2007 Witness Fuminobu Kitayama